

Analysis of butylhydroxytoluene (BHT) in polyethylene (PE) by Thermal Desorption Gas Chromatography (TD-GC)

[Background] When flash pyrolysis is used for the analysis of additives in a polymer, interferences caused by the pyrolyzates of the polymer backbone and/or the pyrolysis of the additives themselves often leads to poor analytical precision. Data quality will dramatically improve by using TD-GC, which utilizes lower temperatures to “extract” the additives of interest. In addition, TD/GC does not require cumbersome sample preparation; the sample is analyzed “as is”. This eliminates errors associated with conventional sample preparation techniques (e.g. solvent extraction). This Technical Note presents the quantitative analysis of BHT (chemical structure and its mass spectrum are shown in Fig. 1.), a common antioxidant additive used in PE using TD-GC/MS.

[Experimental] A piece of the PE sample (1 x 1 x 3mm in size, ca. 3mg) is placed in a sample cup. Note that the shape (surface area to sample weight ratio) is known to affect analytical precision. The ramifications of using “pieces” of sample on data quality will be reported elsewhere(1). The thermogram of a PE sample containing ca. 500ppm of BHT is obtained using evolved gas analysis (EGA): 100-600°C. Extracted ions are used to determine the TD zone for the analysis of BHT.

[Results] The thermogram of the PE sample, obtained by EGA, is shown in Fig. 2. In the TIC chromatogram, only a single peak, emanating from the pyrolysis of PE, is observed. Extracted ion chromatograms: m/z 205 and 220 which are characteristic ions for BHT, are observed in the temperature range 100-200°C. This defines the thermal desorption conditions (100-200°C at 20°C/min, 3 min hold) for determining BHT in PE. Fig. 3 shows a typical chromatogram obtained by TD-GC using these conditions. The peak area of BHT / weight of PE gives a reproducibility less than 3% RSD. The BHT concentration is 510 ppm – based upon an ESTD calibration curve obtained under the same conditions.

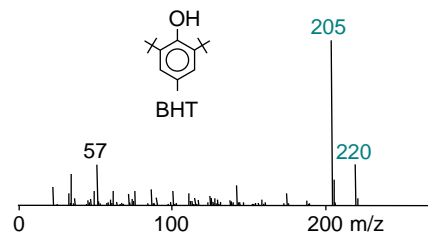


Fig. 1 Chemical structure of BHT and its mass spectrum

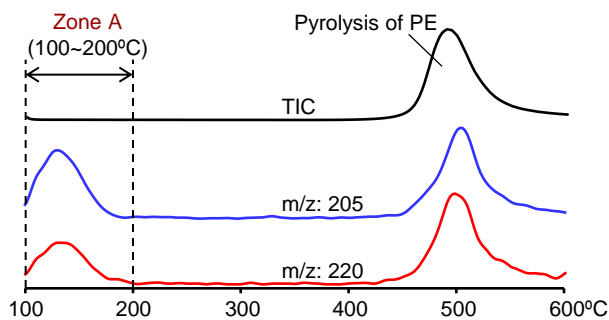


Fig. 2 Thermograms of a PE Sample
Furnace temp. : 100-600°C, (20°C/min), split ratio : 1/50
Sample size : about 0.5mg, detector : MS

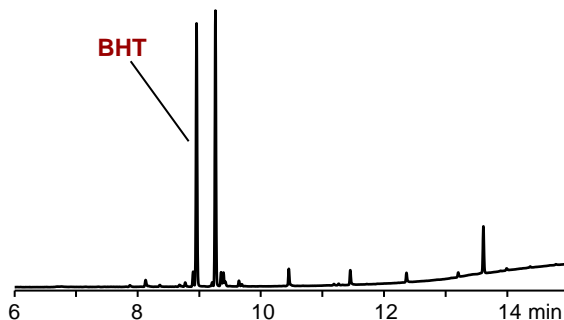


Fig. 3 Chromatogram of thermally desorbed components from a PE sample
Furnace temp.: 100-200°C (20°C/min, 3min hold), flow rate : 1 mL/min, split ratio : 1/50, GC oven temp.: 40-320°C (20°C/min), sample size : about 3mg, detector : FID, separation column : Ultra ALLOY-5 (5%-diphenyl 95%-dimethyl polysiloxane, L=30m, id=0.25mm, df=0.25µm)

Keywords : Additives, Thermal desorption, BHT, Antioxidant, Polyethylene

Products used : Multi-functional pyrolyzer, Auto-Shot Sampler, Vent-free GC/MS adapter, UA-5, Deactivated metal capillary tube

Applications : General polymer analysis

Related technical notes : PYA1-056E

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